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Procedure for Substrate Temperature Control Using the Pyrometer During MBE Growth

Stefan Svensson

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Abstract

Command procedures have been developed for the Army Research Laboratory molecular beam epitaxy (MBE) computer control system that allow a user to automatically outgas and desorb the oxide from substrates before growth, as well as set substrate temperatures based on pyrometer readings during growths. These procedures allow completely unattended growth of structures once suitable temperatures have been determined.

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Background

The Army Research Laboratory molecular beam epitaxy (MBE) system is controlled by a PC-based system that uses the "Molly" software package supplied by EPI. The Molly system provides a script language that can be used to create command procedures that execute customized sequences of actions on the MBE system. Allowed actions are reading and setting cell temperatures, opening and closing shutters, reading pressure gauges, setting the azimuthal rotation speed of the substrate, and turning the substrate holder to the growth and load positions.

Before deposition of material is started, the native oxide on the substrate must be desorbed. This is done by heating the substrate to a predetermined temperature under As overpressure. Due to differences in emissivity of the various substrate holders because of design, size, and age, a specific thermocouple setting cannot be expected to produce the desired temperature with enough precision. Hence, small corrections to the set points must be applied, based on reading of the optical pyrometer.

Many device structures consisting of various material combinations may require different substrate temperatures during the growth sequence. For the reasons mentioned above, it is not possible to preprogram a temperature profile for the thermocouple since it may not result in the desired temperatures as read by the pyrometer. An operator must therefore be present to make the final temperature adjustments. Automation of these adjustments leads to time savings and, in some cases, greater precision and reproducibility.

Script for Outgassing

The logic behind the script developed for substrate outgassing is as follows: The substrate temperature is first raised to a thermocouple set point, at and above which the pyrometer operates (440 °C). Below this temperature the bandgap of GaAs is large enough that the substrate is transparent, which results in a very high pyrometer reading since the pyrometer "sees" the heater elements behind the wafer. After the minimum operation temperature has been reached, the computer starts reading values from the pyrometer. (The critical temperature is considered reached when the pyrometer reading exceeds 450 °C and the time derivative of the temperature is positive.) The pyrometer can be read from either the analog or digital outputs of the instruments. The digital output is greatly preferred due to the

much lower noise level on the signal. (The analog output was used originally because of a malfunction with the digital output port and produced a signal that required heavy averaging and thus more script code.)

The ramp between the minimum operating temperature of the pyrometer and the final outgassing temperature is approximated with a 10-step staircase during which a PID-control (proportional integration and differential) routine drives the pyrometer temperature to each set point. When the final outgassing temperature is reached, it is held constant with software PID control for a specific time, typically 10 min. After this, the temperature is ramped down to a desired growth temperature with the same control algorithm. The outgassing script has now finished and a growth script can be started. The code for the outgassing sequence is called **Genoutg.cmd** and can be used for both GaAs and InP substrates. A parameter flag must be set to provide information about which substrate is used. The control code for the outgassing script is shown in in the appendix (Command File Listings).

Script for Temperature Control During Growth

By running the script **Tsub.cmd** in parallel with the structure recipe during growth, one can change the substrate temperature, as read by the pyrometer, from the structure recipe. Again, PID software control is used. The temperature set point is stored in a global variable (see next section), which is read every 5 s by **Tsub.cmd** and can be changed in the recipe by loading of command files named **TsubXXX.cmd**, where XXX is the temperature. Allowed values are 450 °C < XXX < 700 °C in 5° increments.

Starting the Scripts

The appendix shows an example of a structure recipe for a PHEMT (pseudomophic high-electron-mobility transistor), which will be used to illustrate the start of the scripts. All command procedures are invoked with the Molly command *load*. A few comments about this command are appropriate at this point. The Molly language also includes a command *nsh*, which can "launch" another command procedure. The difference between loading and launching is that a loaded command file must execute to completion before the next step in the calling (loading) file can continue. A launch, on the other hand, starts the new command file and immediately continues with the next step. In other words, the calling and the called procedure will run in parallel. For unknown reasons, the Molly spreadsheet accepts only

the *load* command and not *nsh*. Incidentally, the new EPICAD program allows both *load* and *nsh*.

The first command file, **Stdlog1.cmd** (see appendix), starts a data logger. This is a personal preference of mine and is not needed for the pyrometer control. The same is true for the next command, **Rot5.cmd**, which sets the rotation speed. Next, the outgassing procedure, **Genoutg.cmd**, follows. This should run to completion before the growth begins and is therefore started directly with a *load*.

Since we clearly want to allow the temperature controller and the following structure recipe file to run in parallel, we must load an additional intermediate file, **Pyroc.cmd** (see appendix), which contains only the *nsh* command that launches the desired file (**Tsub.cmd**) (see appendix). This is done immediately after the outgassing.

The temperature set point, which is used in the PID control, is the same as the last temperature in **Genoutg.cmd**. This temperature is stored in the global variable *Tsub_new*, which is declared in the file **C:\molly\lib\local.cmd**, a command file that is executed automatically when the Molly system is started. One can make subsequent changes to the substrate temperature by loading files called **TsubXXX.cmd**, where XXX is the temperature (see appendix). Allowed values are 450 °C < XXX < 700 °C in 5° increments. These command files only set the global variable to a new value, so their execution time is negligible. If a different temperature is desired, any of the existing files can be copied and renamed to the new temperature and the set point edited. (Temperatures below 450 °C should not be used for the obvious reason that the pyrometer does not work in that range.) In the PHEMT recipe, loading **Tsub500.cmd** lowers the temperature from 600 °C, which is the temperature at the finish of the outgassing, to 500 °C just before the InGaAs layer is started.

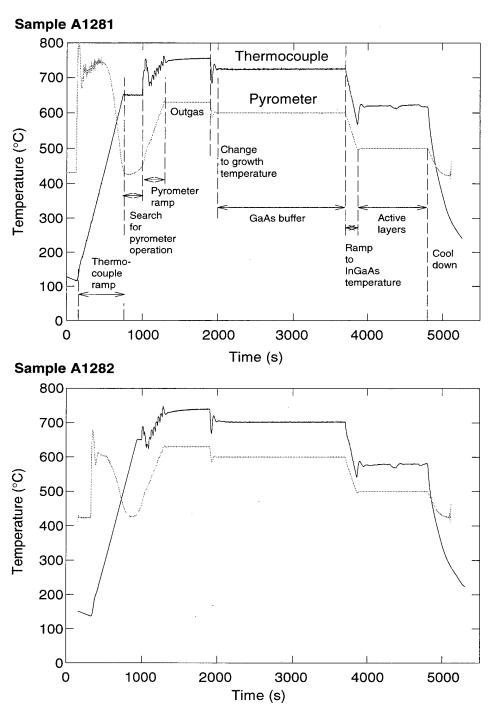
Before the InGaAs growth starts, another GaAs layer is inserted after the **Tsub500.cmd**. This illustrates an important limitation of the PID routine. If the temperature change is too large, an abrupt change in set point may result in a substantial under- or overshoot. An undershoot can be particularly problematic since it may drive the substrate temperature below the operating range of the pyrometer. To avoid this, the **Tsub.cmd** file always applies a ramp with a rate of 40°/min. Hence, in the PHEMT example, the new set point, which is 100 °C below the previous one, will not be reached until 150 s after **Tsub500.cmd** is loaded, requiring the extra GaAs layer (180 s is used in the recipe for extra margin).

When the recipe has finished, the **Tsub.cmd** should be terminated. This is done by loading **Kill_pyr.cmd** (appendix). The latter executes the command *kill(pyro_id, SIGKILL)* in which *pyro_id* is another global variable defined in **C:\molly\lib\local.cmd**. Similarly, the logging is stopped with the file **Kill_log.cmd** (appendix). The files **Rot0.cmd** and **Pos1.cmd** turn off the azimuthal rotation and turn the substrate to the loading position. (Note

that the pyrometer control definitely *must* be turned off before the substrate is turned in another direction.)

Figure 1 shows the results of two consecutive growths (run numbers 1281 and 1282) with the automated outgassing and the change of substrate temperature during deposition. Despite the fact that two different types of substrate holders (EPI UNI-Block and Varian nonbonded) were used, the pyrometer traces are virtually identical beyond the outgassing phase.

Figure 1. Temperature as a function of time during growth of two consecutive PHEMT wafers. Solid trace represents thermocouple data and dotted trace represents pyrometer temperature.



Final Comments

The files Genoutgas.cmd, Tsub.cmd, TsubXXX.cmd, and Pyroc.cmd are all stored in C:\u\mbe\. This allows the use of the file name only (without the explicit path name) in the growth recipe. As mentioned above, the global parameters are created in C:\molly\lib\local.cmd.

The parameters that are coded into the script files all have comments next to their declarations. It should be fairly obvious from the script listings what the parameters represent. Some optimization has been done and the present values do work. However, further optimization can probably be done and certainly refinements are possible. In particular, I am planning to allow a step change in temperature if the difference between the old and new set points is below a certain value. Once I am satisfied that the outgassing routine works perfectly for both InP and GaAs, I will probably create separate files so that the operator does not have to edit the parameter *SubToday*.

Acknowledgment

I used the PID controller, coded and optimized by Rich Leavitt.

Appendix. Command File Listings

PHEMT8.wb1 Molly spreadsheet recipe for a PHEMT

Genoutgas.cmd General outgassing procedure for GaAs and InP

Stdlog1.cmd Starts the standard logging procedure

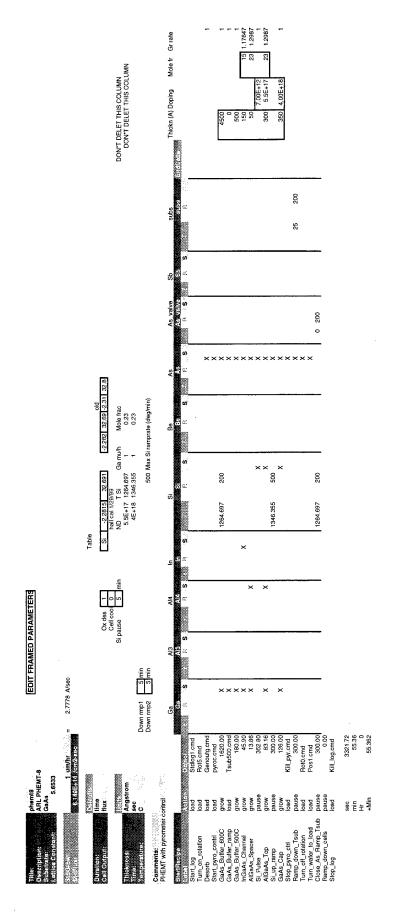
Pyroc.cmd Launches Tsub.cmd

Tsub.cmd General PID controller for MBE growth

Tsub550.cmd Changes the pyrometer set point to 550 °C

Kill_pyr.cmd Terminates Tsub.cmd

Kill_log.cmd Terminates the data logging



24-Jul-2000 phemt8 Auto oxide desob - Variable Growth Cells ramp down - Variable

Genoutgas.cmd Appendix

```
/*
   This command file desorbs the oxide from an substrate which can be either
   GaAs or InP (not tested yet)
   Temperature control via pyrometer and PID routine from Rich Leavitt
                                             Stefan Svensson, ARL Mar 9, 2000
/**/
#include <stdlib.h>
#include <signal.h>
#include <unistd.h>
#include <cells.h>
#include <gauges.h>
#include <shutters.h>
#include <time.h>
#include <mbe.h>
/**/
/*
                                             -TPoutgas
          TC work-
             Ramp_time1 Ramp_time2 Ramp_time3
                |--| |-----
                Wait_time1 Outg_time
                 | Pyro reading starts
/**/
                                 /* Substrate flag
double Substrate;
                                 /* GaAs
double GaAs
                       = 1;
double InP = 0; /* InP
double SubToday = 1; /* Today's substrate
double TPoutgasInP = 550.0; /* Pyrometer Max outgassing temp InP
double TPoutgasGaAs = 630.0; /* Pyrometer Max outgassing temp GaAs
                                                                                                         */
*/
double TPoutgas;
                                   /* Pyrometer Max outgassing temp General
 double TP_growInP = 525.0; /* Pyrometer Growth temperature InP
                                                                                                         */
*/
 double TP_growGaAs = 600.0; /* Pyrometer Growth temperature GaAs double TP_grow; /* Pyrometer Growth temperature General
 double TP_grow;
                                                                                                         */
                                   /* Pyrometer Difference between last two readings
 double TPdelta;
                                                                                                         */
*/
*/
                          /* Pyrometer general substrate temperature
                                   /* Pyrometer Previous reading
 double TPlast;
 double TPsub;
                       /* Pyrometer reading at start of PID control ramp

= 440.0; /* Pyrometer min real temperature

/* Pyrometer step length in PID control ramp
 double TP0;
                                                                                                         */
 double TPwork
                                                                                                         */
 double TPstep;
                                                                                                         */
                                   /* Thermocouple general substrate temperature
 double TCsub;
                                  /* Thermocouple Min operating temp (first ramp target)
 double TC work;
                                                                                                          */
 double TC_workGaAs = 650.0; /* Thermocouple Min operating temp GaAs
 double TC_workInP = 500.0; /* Thermocouple Min operating temp InP
double TC_test = 450.0; /* Thermocouple start testing for pyrometer operation
                                   /* Thermocouple Ramp rate
 double Ramp_rate;
double Ramp_time1 = 10.0; /* Minutes ramp time to temp where pyrometer works double Ramp_time2 = 5.0; /* Minutes ramp time to outgassing temp double Ramp_time3 = 3.0; /* Minutes ramp time to outgassing temp to double Wait_time1; /* Minutes to wait for pyrometer to start working double Wait_time2 = 0.1; /* Minutes to wait between pyrometer readings double Wait_time3 = 0.5; /* Minutes extra wait before pyrometer control
```

```
double Outg_time
               = 10.0; /* Minutes
                                 outgas time
double Time_step;
                      /* Time step length in ramp
double Numb_steps
               = 10.0; /* Number of steps that ramp is broken into
                      /* Counter for pyrometer readings
int
int
                      /\star Number of pyrometer readings to average
               = 5;
     I_pyro
double
 t0.
  control time,
 Tstep = 1,
 sigma,
 delta,
 Prop = 72,
 Integ = 4.7,
 Deriv = 1.35,
 Tmin = 25,
 Tmax = 850,
 errtot=0.
 TCsetp,
 oldT,
 newT,
 curr_err,
 prev_err;
string sznewT, szct;
/**/
/\star========= Initialize parameters for the right substrate =============*/
/**/
if(SubToday == GaAs)
   TPoutgas = TPoutgasGaAs;
   TP_grow = TP_growGaAs;
   TC_work = TC_workGaAs;
if (SubToday == InP)
   TPoutgas = TPoutgasInP;
   TP grow = TP_growInP;
   TC_work = TC_workInP;
echo(" TPoutgas = ", TPoutgas);
echo(" TP_grow = ",TP_grow);
echo(" TC_work = ",TC_work);
/**/
/*=========== the pyrometer starts working
                                         =========*/
/**/
while ( TCsub <= 0.0 ) TCsub = temp( subs );
                                      /* Read current TC set point
Ramp_rate = fabs( (TC_work - TCsub )/Ramp_time1);
set_ramp( subs ,Ramp_rate );
                                       /* Ramp thermocouple
set_temp( subs ,TC_work);
                                       /*
                                             to temp where pyro works */
echo(" Ramping substrate to pyrometer range (Thermo-couple= ",TC_work,")");
echo(" This will take ", Ramp time1," min."
                                 );
echo("");
                                       /* Wait until ramp is done
                                                                 */
echo(" Waiting for substrate to thermalize and pyrometer to start working ");
Wait_time1 = Ramp_time1*1.1;
sleep ( Wait_time1*60);
                                       /* Start checking pyro just before*/
                                          ramp finsihes
```

Genoutgas.cmd Appendix

```
/*======== Start testing for pyrometer operation ========================= */
/**/
echo(" Looking for pyrometer stability ");
                                       /* initialize
TPdelta = -1.0;
                                       /* Search for positive TPdelta
TPlast = 1000.0;
TCsub = 0.0;
TPsub =
         0.0;
while (TPdelta <0.0 || TPsub < TPwork)
   i=0;
   TPsub = 0.0;
   while (i < I_pyro)
     i=i+1;
      TPsub = TPsub + reading(pyrometer);
             echo(" i, TPsub =",i,TPsub); */
/*
      sleep(0.5);
   TPsub = TPsub/I_pyro;
   TPdelta = TPsub - TPlast;
   TPlast = TPsub;
           echo(" TPsub, TPdelta = ", TPsub, TPdelta); */
   sleep ( Wait time2*60);
                                       /* Wait an extra 30 sec (chicken!) */
sleep ( Wait_time3*60);
/*-----*
echo(" Starting pyrometer control and upramp");
echo("");
                                             /* Time per step in sec */
        = Ramp_time2*60.0/Numb_steps;
Time_step
control_time = Time step;
TPO = reading(pyrometer);
        = (TPoutgas - TP0)/Numb_steps;
echo(" Current temperature = ",TPO);
/* echo("control_time = ",control_time);
/* echo("TPstep = ",TPstep);
/* echo("Tstep = ",Tstep);
        = 0;
while (i < Numb steps)
     {
           = i+1;
     i
     newT = TPstep*i + TP0;
     echo("New temp:",newT);
     sigma = -setp(subs)*Integ*Prop/100;
     prev_err = reading(pyrometer)-newT;
     set_ramp(subs,1000);
     oldT
          = newT;
           = t;
     tm
     t0
           = t;
     while ( t-t0 < control_time )
          sleep(Tstep - (t - tm));
          curr_err = reading(pyrometer)-newT;
          sigma = sigma+Tstep*curr_err;
          errtot = errtot+Tstep*curr_err*curr_err;
          delta = (curr err-prev err)/Tstep;
          TCsetp = -(curr_err+sigma/Integ+delta*Deriv)*100/Prop;
          if (TCsetp < Tmin) TCsetp = Tmin;
          if (TCsetp > Tmax) TCsetp = Tmax;
          set temp(subs, TCsetp);
          prev err = curr_err;
```

```
tm = tm + Tstep;
         tm=t;
/**/
/*-----*/
echo(" Outgas temperature reached ");
echo(" Outgassing for ", Outg_time, " min");
    control_time = Outg_time*60.0;
    newT
           = TPoutgas;
/*
     echo("New temp:",newT);
    sigma = -setp(subs)*Integ*Prop/100;
    prev_err=reading(pyrometer)-newT;
    set_ramp(subs, 1000);
    oldT
            = newT;
             = t;
    tm
    t0
            = t;
    while ( t-t0 < control_time )</pre>
         sleep(Tstep - (t - tm));
         curr_err = reading(pyrometer)-newT;
         sigma = sigma+Tstep*curr_err;
         errtot = errtot+Tstep*curr_err*curr_err;
         delta = (curr_err-prev_err)/Tstep;
TCsetp = -(curr_err+sigma/Integ+delta*Deriv)*100/Prop;
         if (TCsetp < Tmin) TCsetp = Tmin;
         if (TCsetp > Tmax) TCsetp = Tmax;
         set_temp(subs,TCsetp);
         prev_err = curr_err;
            = tm + Tstep;
         tm
               = t;
         tm
         }
/**/
/**/
echo("");
echo(" Done outgassing ");
echo(" Starting ",Ramp_time3," min down-ramp");
echo("");
control_time = Ramp_time3*60;
TP0 = reading(pyrometer);
echo("Current temperature = ",TPO);
    newT
          = TP_grow;
    echo("New temp:",newT);
    sigma = -setp(subs)*Integ*Prop/100;
    prev_err = reading(pyrometer) -newT;
    set_ramp(subs, 1000);
        = newT;
    OldT
    tm
          = t;
    t0
          = t;
    while ( t-t0 < control_time )</pre>
        sleep(Tstep - (t - tm));
        curr_err = reading(pyrometer) -newT;
        sigma = sigma+Tstep*curr_err;
        errtot = errtot+Tstep*curr_err*curr_err;
        delta = (curr_err-prev_err)/Tstep;
TCsetp = -(curr_err+sigma/Integ+delta*Deriv)*100/Prop;
        if (TCsetp < Tmin) TCsetp = Tmin;</pre>
        if (TCsetp > Tmax) TCsetp = Tmax;
```

Genoutgas.cmd Appendix

```
/* This command procedure starts a data log of temperatures,
/\star~ shutter positions and the flux gauge during a growth.
/* The data is stored in the file
    C:/u/mbe/recipes/stefan/log_data/outfile.dat
                                                /* log every 20 seconds */
log_id = logger(20.0,
             `t',
             'temp(subs)',
             'temp(Ga)',
             'temp(Al3)',
             'temp(A14)',
             'temp(In)',
             'temp(Si)',
             'temp(Be)',
             'temp(Sb)',
             'temp(As)',
             'temp(As_valve)',
             'is_open(Ga)',
             'is_open(Al3)',
'is_open(Al4)',
             'is_open(In)',
             'is_open(Si)',
             'is_open(Be)',
             'is_open(Sb)',
             'is open(As)',
             'reading(flux)',
             "c://u//mbe//recipes//stefan//log_data//outfile.dat");
echo("log_id = ",log_id);
```

Pyroc.cmd Appendix

Appendix Tsub.cmd

```
******************
   This command file sets a new substrate temperature over a time interval of
   50000 seconds (in other words it runs until you kill it).
/* New setpoints are entered by setting the global variable Tsub_new in an other
   command file. Enter the line load Tsubxxx.cmd (where xxx is the temp)
  Temperature control via pyrometer and PID routine from Rich Leavitt
/*
/*
/*
                               Stefan Svensson, ARL Jun 09, 2000
#include <stdlib.h>
#include <signal.h>
#include <unistd.h>
#include <cells.h>
#include <gauges.h>
#include <shutters.h>
#include <time.h>
#include <mbe.h>
double recipe_time = 50000.0;/* Seconds total time the cmd file is running
                       /* Seconds start time for cmd file
                       /* Pyrometer reading at start of PID control ramp
/* Pyrometer step length in PID control ramp
double TP0;
double TPstep;
                       /* Thermocouple general substrate temperature
double TCsub;
double TCsub; /* Thermocouple general substrate temperature double TC_test = 450.0; /* Thermocouple start testing for pyrometer operation
                                                                      */
*/
*/
*/
double Ramp_rate = 40; /* Thermocouple Ramp rate (deg/min)
                       /* Time step length in ramp
double Time_step;
double Numb steps = 10.0; /* Number of steps that ramp is broken into
                       /* Counter for pyrometer readings
int i;
int I_pyro
                                                                      */
*/
               = 5; /* Number of pyrometer readings to average
                      /* Pyrometer New temperature value used inside PID loop
/* Seconds Start time for PID loop
double newT;
                                                                       */
double t0;
                  /* Seconds duration of PID loop
                                                                      */
double control_time;
double Tstep = 1.0; /* Seconds wait time between PID steps
                                                                      */
                      /* PID sigma
double sigma;
double delta; /* PID delta
double Prop = 72.0; /* PID prop constant
double Integ = 4.7; /* PID integration constant
double Deriv = 1.35; /* PID derivative constant
double Tomin = 25.0 /* PID derivative constant
                                                                       */
                                                                       */
                       /* PID integration constant
                                                                      */
                                                                      */
              = 25.0; /* Thermocouple min allowed value
double TCmin
              = 850.0; /* Thermocouple min allowed value
                                                                       */
double TCmax
               /* Thermocouple setpoint
= 0.0; /* PID total error
                                                                       */
double TCsetp;
double errtot
double curr_err;
                      /* PID current error
                      /* PID previous error
double prev_err;
/*------
/**/
                                /* Start time */
t00
      = t;
while ( t-t00 < recipe_time )</pre>
    {
     echo("First test line, Tsub_new = ",Tsub_new); */
     if( Tsub_old != Tsub_new )
/*-----*
```

Tsub.cmd Appendix

```
/**/
       echo(" Starting pyrometer control to ", Tsub_new );
                 = reading(pyrometer);
       TPO
                 = (Tsub_new - TP0)/Numb_steps;
       TPstep
       Time_step
                                             /* Time per step in sec */
                 = fabs(TPstep)/Ramp_rate*60.0;
       control_time = Time_step;
                = 0;
        echo(" Current temperature = ",TPO); */
/**/
       while (i < Numb steps)
            {
                   = i+1;
            i
                   = TPstep*i + TP0;
            newT
                                        echo("New temp:", newT);
                   = -setp(subs)*Integ*Prop/100;
            prev_err = reading(pyrometer)-newT;
            set_ramp(subs,1000);
                  = t;
            t0
                   = t;
            while ( t-t0 < control_time )</pre>
                   sleep(Tstep - (t - tm));
                   curr_err = reading(pyrometer)-newT;
                          = sigma+Tstep*curr_err;
                   errtot = errtot+Tstep*curr_err*curr_err;
                   delta = (curr_err-prev_err)/Istep;
TCsetp = -(curr_err+sigma/Integ+delta*Deriv)*100/Prop;
                   if (TCsetp < TCmin) TCsetp = TCmin;</pre>
                   if (TCsetp > TCmax) TCsetp = TCmax;
                   set temp(subs,TCsetp);
                   prev_err = curr_err;
                   tm = tm + Tstep;
                   tm=t;
       echo(" New substrate temp reached ", newT);
       Tsub old = Tsub_new;
               */
          else
/**/
         echo(" Maintaining temp at =",Tsub_new);
/**/
                                              /*
                                                 Control for 5 sec before
        control_time = 5.0;
                                              /*
                 = Tsub_new;
                                                 testing for new Tsub
                 echo("New temp:",newT);
                   = -setp(subs)*Integ*Prop/100;
         sigma
                  = reading(pyrometer)-newT;
        prev_err
        set_ramp(subs,1000);
                  = t;
        t0
                  = t;
        while ( t-t0 < control_time )
             sleep(Tstep - (t - tm));
             curr_err = reading(pyrometer)-newT;
                                   echo("newT = ",newT); */
/*
                     = sigma+Tstep*curr err;
             sigma
             errtot = errtot+Tstep*curr_err*curr_err;
             delta = (curr_err-prev_err)/Tstep;
             TCsetp = -(curr_err+sigma/Integ+delta*Deriv)*100/Prop;
                 if (TCsetp < TCmin) TCsetp = TCmin;
```

```
if (TCsetp > TCmax) TCsetp = TCmax;
set_temp(subs,TCsetp);
prev_err = curr_err;
tm = tm + Tstep;
tm = t;
}
```

Tsub550.cmd Appendix

Kill_log.cmd Appendix

Distribution

Admnstr Defns Techl Info Ctr Attn DTIC-OCP 8725 John J Kingman Rd Ste 0944 FT Belvoir VA 22060-6218

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Command procedures have been developed for the Army Research Laboratory molecular beam epitaxy (MBE) computer contról system that allow a user to automatically outgas and desorb the oxide from substrates before growth, as well as set substrate temperatures based on pyrometer readings during growths. These procedures allow completely unattended growth of structures once suitable temperatures have been determined.				
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